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# Shear history effect on the viscosity of carbon nanotubes water-based nanofluid

Patrice Estellé <sup>a</sup>, Salma Halelfadl <sup>a,b</sup>, Nimet Doner <sup>c</sup>, Thierry Maré <sup>b</sup>

<sup>a</sup> LGCGM EA3913, Equipe Matériaux et Thermo-Rhéologie, Université Rennes 1, IUT de Rennes, 3 rue du Clos Courtel, BP 90422, 35704 Rennes Cedex 7, France

<sup>b</sup> LGCGM EA3913, Equipe Matériaux et Thermo-Rhéologie, Université Rennes 1, IUT de Saint-Malo, Rue de la Croix Désilles, CS51713, 35417 Saint-Malo Cedex, France

<sup>c</sup> Department of Mechanical Engineering, Dumlupinar University, 43270 Kutahya, Turkey

\* Author to whom correspondence should be addressed.

Electronic mail: [patrice.estelle@univ-rennes1.fr](mailto:patrice.estelle@univ-rennes1.fr)

Tel: +33 (0) 23 23 42 00

Fax: +33 (0) 2 23 23 40 51

**Abstract:** Experimental results on the steady state rheological behaviour of carbon nanotube (CNT) water-based nanofluid are presented. We have investigated the influence of a controlled preshear history on the viscosity of CNT water-based nanofluid. Two types of preshear history effect are studied: the influence of stress rate during preshear and the effect of resting time before viscosity measurement. It is revealed that CNT water-based nanofluid behaves as a viscoelastic media at low shear rate and it is shear-thinning at higher shear rate. This behaviour is strongly dependent on shear history due to the breakdown in the structural network of nanofluid agglomerates. It is also observed that the nanofluid can reform at rest after preshear following the resting time and the rate or preshear applied to the nanofluid.

**Keywords:** agglomerates, CNT, nanofluid, preshear history, resting time, shear-thinning, viscosity

## **Graphical abstract**

Rheological behavior of carbon water based nanofluid is dependant on shear history effect due to the breakdown in the structural network of nanofluid aggregates.

## 1. Introduction

Nanofluids typically consist of nanometer-sized particles of metals, oxides, nitrides, carbides or carbon nanotubes dispersed in a conventional base fluid such as water, glycol, ethylene glycol, engine oil... Nanofluids constitute a class of colloidal suspensions with enhanced thermal properties compared to the base fluids even at under low concentrations of suspended nanoparticles. Previous studies have shown the complex feature of nanofluid rheology.

Whereas conventional base fluids mainly behave in Newtonian manner, adding nanoparticles to a base fluid can only alter the magnitude of viscosity [1-5]. It can also result in different types of rheological behaviour, namely shear-thinning [4;6-16] and yield stress material [7, 17-19] depending on nanofluid. Moreover, viscoelastic behaviour is also observed [20-22]. These investigations have revealed that the rheological behaviour of nanofluids is strongly affected by the viscosity of the base fluid, the nanoparticle shape, the particles size and concentration, the temperature and the shear rate. The rheological properties of nanofluids are also closely related to the dispersion state of the nanosuspensions, their stability and the presence (or not) of agglomerates. So, it is clear that shear-thinning behaviour of nanofluids is mainly associated with the presence of nanoparticle agglomerates even with Newtonian base fluids [8-10, 23-25].

There are two methods for dispersing nanoparticles within the base fluid and formulating a stable nanofluid [26,27]. The first one is based on the physical or chemical treatment of the nanoparticles, in particular for carbon nanotubes [28] and the use of dispersing agents such as polymers. The dispersing of nanoparticles can also be obtained from mechanical methods such as sonication [2,4,17,21], ultrasonication [3,5-7,12], homogenization [6,12] and high stirring [3,5,14]. The dispersion and the stability of the nanofluids are mainly obtained by gathering some of the previous mentioned methods. So, at sufficient rate and duration of mechanical mixing, it is expected that the agglomerates are broken and the nanoparticles are well dispersed. However, it should be mentioned that mechanical methods are able to reduce the size of aggregates but not sufficient to obtain a complete dispersion of the nanoparticles into primary particles [10,13]. In addition to this, it was reported that after passing the optimized duration of mechanical dispersing, the nanofluid can agglomerate in a clog during a fast sedimentation [29]. With nanotubes, high mechanical dispersion can lead to the breaking of the nanotubes and results in shorter nanotubes [30].

A number of studies related to the effect of dispersing mechanical methods on the viscosity of nanofluids have been reported. Duan et al. [5] have investigated the effect of ultrasonification of the rheological behaviour of  $\text{Al}_2\text{O}_3$  water nanofluid for volume concentration range of 1-

5%. It was shown that the nanofluids, which initially behave as shear-thinning material due to the presence of agglomerates, resumed as Newtonian fluids just before ultrasonification. Garg et al. [30] have reported the effect of sonication on the viscosity of CNT nanofluid. They have shown that sonication time is first associated with declustering of nanoclusters. Then, increasing the sonication time leads to breakage of the nanotubes resulting in less pronounced shear-thinning behaviour because of the shorter size of the nanotubes. Yang et al. [7] have also shown the effect of frequency and time of ultrasonication on agglomerate size and aspect ratio of nanotubes dispersed in oil. They have shown that aspect ratio of the nanotubes decreases as both the dispersing time and energy increase, resulting in less viscous nanosuspensions.

Based on the previous observations, the shear history of a nanofluid must be taken into consideration when investigating its rheology. In this paper, we studied experimentally the effects of preshear history on the viscosity of a CNT water-based nanofluid with a nanotubes weight fraction of 1%. Rheological behaviour under steady-state condition after a well-controlled preshear of the nanosuspension has been investigated. Moreover, the effect of preshear stress rate and resting time on the observed rheological behaviour is presented and discussed in terms of structure and stability of the nanofluid.

## **2. Materials and methods**

### **2.1 Nanofluid**

A carbon nanotube water-based suspension was prepared and provided by Nanocyl (Belgium). This suspension consists on multi-walled carbon nanotubes (carbon purity 90%) dispersed from ultrasonication in a base mixture of de-ionized water and sodium dodecyl benzene sulfonate (SDBS) as surfactant. The properties of nanotubes and nanofluid suspension presently used are presented in Table 1 and Table 2 respectively. The dimensions of the nanotubes are 1.5  $\mu\text{m}$  in average length  $L$  and 9.2 nm in average diameter  $d$  respectively. This leads to an average aspect ratio  $r=L/d\approx 163$ . According to the manufacturer's specification, the nanofluid was stable for several months.

As mentioned before, the surfactant is used to disperse and stabilize the particles and reduce the presence of aggregates because of the hydrophobic surface of carbon nanotubes [28]. However, it was previously reported that carbon nanotubes suspension can form clusters in spite of the use of surfactant. So, SEM characterization of the studied nanofluid was

performed to investigate the dispersion state of the nanoparticles within the base mixture. A droplet of the nanofluid was preliminary dried then covered by gold before SEM analysis. The SEM characterization was conducted on a LEO 1430 VP model SEM apparatus with tungsten filament at an accelerating voltage of 15 kV. The structure of the nanofluid observed by SEM is shown in Figure 1. The observations from Figure 1 indicate the presence of aggregate network within the nanofluid. The approximated size of the aggregates varies between 1 nm, which is close to the average length of the nanotubes, and 6 nm.

It should be finally mentioned that any observable sedimentation was noticed before experiments. Noting also that the CNT nanofluid was used as received and no mechanical pretreatment such as sonication was applied to the nanofluid before the SEM characterization and the rheological measurements.

## **2.2. Rheological measurements**

Shear viscosity of CNT nanofluid was measured by using a stress controlled rheometer (Malvern Kinexus Pro) in a parallel plate configuration under a controlled temperature of 20°C. The temperature was controlled by using a peltier temperature control device located below the lower plate. Thermal clovers were also used to ensure constant temperature within the sample gap. All experiments are conducted with a 40mm plate diameter and under a constant gap of 0.5mm. The shear viscosity of the base mixture was also measured under the same experimental conditions.

Each tested volume sample was taken from its container with a syringe-type automatic pipette then set up to the lower plate. Hence, the upper plate is displaced to achieve the required sample gap. The excess of samples is eventually removed. An initial stabilization period of 5 min is given for achieving constant working temperature with an accuracy of 0.01°C. This temperature was also maintained for 5 minutes before starting the experiment. Consequently, each sample has the same shear history before being used in the rheological experiments. A new sample was used for each measurement which was repeated to evaluate the repeatability of the measurement. No difference was obtained indicating that the nanofluid is stable and homogeneous before testing it.

Before the measurement of the nanofluid steady shear viscosity, the experiment was first started by applying a constant shear stress from the rheometer for 60s. This preshear stress time is sufficient to approach a quasi-equilibrium state. The used shear stress values for preshear were set to 0.5, 1, 3, 5, 7 and 10Pa respectively. Hereafter, without any resting time,

to ensure the results were not affected by a structural evolution at rest of the nanosuspension after pre-shearing, the effect of this preshear history on the nanofluid viscosity was measured by conducting a steady stress logarithmic sweep test over the stress range from 0.1Pa to 10Pa. Another series of experiments were performed setting at rest the presheared nanofluid sample before measuring the nanofluids steady-state shear viscosity. This was done to evaluate the structural evolution and the aging of the nanosuspension during resting time. The resting times used were 5, 10, 20 and 60min.

The applicability of the shear stress range for the steady stress logarithmic sweep test was preliminary evaluated in order to ensure steady-state flow at low shear stress, and to avoid flow instability and sample ejection at high shear stress. The maximum step duration is 120s. It was observed that apart at the starting of the experiment, a steady-state flow is rapidly achieved and maintained 10s before measuring the viscosity.

De-ionized water and standard oil for calibration were preliminary tested at 20°C showing, as expected, a Newtonian behaviour within the shear stress range investigated. The viscosity value of distilled water and oil was respectively 1.03 and 1360 mPa.s. The relative deviation in comparison to the theoretical values at 20°C is respectively less than 3 and 4%, which is of the same order of magnitude as the experimental uncertainty. The shear viscosity of the base mixture, composed of de-ionized water and sodium dodecyl benzene sulfonate, was reported in Figure 2. It is shown that the base mixture behaves in a Newtonian manner as the shear viscosity is quite constant within the shear rate range investigated. The shear viscosity value of the base mixture is 1.13mPa.s. This is higher than the viscosity of de-ionized water due to the presence of SDBS.

The influence of preshear stress history is investigated in the following, considering the effects of preshear stress rate applied to the nanofluid and resting time before evaluating the viscosity of the nanofluid in function of shear rate and the variation of the network structure of the nanosuspension under shear flow.

### **3. Results and discussion**

#### **3.1 Effect of stress rate during preshear**

Figure 2 shows the apparent viscosity of the CNT nanofluid as a function of shear rate under no preshear history and under seven preshear stress rates. Before considering the effect of preshear history, it is first observed that the curve obtained without preshearing the sample is

above the curves displayed after preshear. This indicates that the set-up of the sample to the lower plate of the rheometer does not disrupt the structure of the CNT nanofluid. So, the curves obtained from different preshear stress rates are representative of the destructure rate of the CNT nanofluid before viscosity measurement.

The curves displayed in figure 2 first reveal that the apparent viscosity increases when the shear stress (or shear rate) increases. This shows a dominant viscoelastic behavior of the CNT suspension due to the interconnected network of agglomerates. Then, once a critical value of shear stress is reached, namely the yield stress, the CNT starts to flow as a shear-thinning suspension, as the apparent viscosity decreases when the shear rate increases. As previously reported and mentioned in the introduction, this behavior is related to the presence of aggregates network within the nanofluid.

The displaying of the curves of figure 2 can be explained as follow. At low shear rate, the aggregates are connected and deform under shear, the nanosuspension does not relax the stress and the suspension resists to flow. Then, at a sufficient shear stress, some interconnections between clusters broke down and de-agglomeration and alignment of the nanotube clusters start to produce resulting in both less viscous force and shear thinning effect when the shear stress increases.

The viscoelastic behaviour of the suspension is strongly dependent on the rate of the applied pre-shear. It is observed from figure 2 that an increase in shear stress level during preshear of the sample results in a less viscoelastic behaviour of the nanofluid as the magnitude of viscosity at low shear rate is lower. This indicates that the higher preshear stress disrupts more the elastic network of the CNT nanoclusters than at lower preshear stress.

It is also mentioned that the shear-thinning behavior of the CNT nanofluid is strongly affected by an increase of preshear stress rate. Higher the preshear stress value, lower the shear-thinning behavior and the extent of the shear-thinning region. This shows the effect of preshear history on the network modification of the nanotube clusters.

It is shown that after a high preshear of 10Pa, the CNT nanofluid tends to behave as a Newtonian fluid, indicating that the structural network of the nanofluid are broken down. It is also shown that the high shear viscosity of the CNT nanofluid is not affected by the preshear history, as the high shear viscosity is similar for the different curves. This also means that the high preshear of the suspension plays a role only in breaking the agglomerates network, not in reducing the size of nanoclusters and nanotubes.

The main findings reported so far can be highlighted by figure 3. In this figure is reported the apparent shear viscosity of the nanofluid for two shear stress value, 1Pa and 10 Pa



respectively, in function of the preshear stress values. It is observed from figure 3 than the apparent viscosity at 1Pa strongly decreases as the preshear stress value increases, due to the network modification of the nanotube clusters following the rate of preshear. Figure 3 also shows than the apparent shear viscosity at a high shear stress of 10Pa is independent of preshear stress rate, and only depends on the final value of applied shear stress during the measurement and the size of aggregates. The shear viscosity of the CNT water based nanofluid at high shear rate (or shear stress) was finally evaluated to 5.8mPa.s. This is higher than the viscosity of the base mixture due to the presence of nanotubes under the form of aggregates.

This can be proven considering the Maron-Pierce viscosity model [31] which derives from the Krieger and Dougherty equation [32] (see equation 1). Noting that equation 1 was obtained from a minimum principle applied to the energy dissipated by viscous effects. These equations were developed for hard spheres systems but have also been used to model experimental data of dispersions with rod like particles [9, 10, 33, 34].

$$\mu_{nf} = \mu_{bm} \left( 1 - \frac{\phi}{\phi_m} \right)^{-2} \quad (1)$$

In equation 1,  $\mu_{nf}$  is the viscosity of the nanofluid and  $\mu_{bm}$  the viscosity of the base mixture, and  $\phi$  is the volume fraction of nanoparticle in base mixture.  $\phi_m$  is the maximum volume fraction of nanoparticle and depends on aspect ratio of the nanotubes [35]. A value of  $\phi_m \approx 3.61\%$  is here obtained due to the very large aspect ratio of the nanotubes presently used.

Comparison of the experimental data of viscosity with the predicted data from the above equation is shown in Figure 3. It is observed that the experimental viscosity values at high shear rate are underpredicted by equation (1). This confirms the presence of aggregates which increase the effective volume of the nanotubes and thus the viscosity of the nanofluid at high shear rate.

With the presence of aggregates, the application of the fractal concept [36] can be used to predict the viscosity of nanofluid at high shear rate. Considering that the geometry of the aggregates can be described as fractal like structure with a fractal index  $D$ , and assuming that the aggregates density change with the radial position and it is not uniform in the nanofluid [8], an effective volume fraction of nanoparticles, denoted  $\phi_a$ , can be defined following equation 2.

$$\phi_a = \phi \left( \frac{a_a}{a} \right)^{3-D} \quad (2)$$

Where  $a_a$  and  $a$  are the aggregates and primary nanoparticles radii respectively.

This leads to the modified Maron and Pierce equation [37]

$$\mu_{nf} = \mu_{bm} \left( 1 - \frac{\phi_a}{\phi_m} \right)^{-2} \quad (3)$$

The fractal index  $D$  depends on the type of aggregation, particle size and shape and shear flow condition. For aggregating nanofluids with nanorods or nanotubes,  $D$  varie between 1.5 and 2.45 [34]. A value of 2.1 is generally taken [9,38,39] and was also used here for the fractal index value.

Figure 3 shows that a good agreement between the prediction of the modified model of Maron & Pierce and experimental data of viscosity at high shear stress (or high shear rate) is obtained when  $a_a/a \approx 4.41$ . Considering the primary nanoparticles radii,  $a$ , as the average lenght of the nanotubes, this leads to a maximum aggregates size close to 6.6 nm, which is in aggrement with the maximum size of aggregates determined from SEM analyses.

### 3.2 Effect of resting time after preshear

Based on the previous results concerning the effects of preshear rate of the rheological behaviour of the nanosuspension, the effect of resting time on the viscosity of presheared nanosuspension was evaluated considering only two rates of preshear stress, 5 and 10Pa respectively. This was done because we had observed that for a preshear of 5Pa, the structure of the nanofluid is partially broken before applying the shear stress sweep. On the other hand, for a preshear of 10Pa, it was shown that the network of nanoclusters is well broken.

Figure 4 shows the apparent viscosity of the CNT water based nanofluid as a function of shear rate after different resting times and an initial preshear stress level of 5Pa.

For lower resting times of 5 and 10min, the curves are superimposed, indicating that the network of the nanofluid does not evolve at rest. It is observed that the maximum shear viscosity at low shear rate slowly increases after a resting time of 20min. This increase is more pronounced for a resting time of 60min. This shows that the nanosuspension structure

build up slowly at rest after cessation of pre-shearing and that the agglomerates reform. Such behavior shows the reversible nature for the rebuild and the association process of the nanoclusters. Our results show that the pre-sheared nanofluid is here not stable, due to the partial breaking of its network structure during pre-shear.

The curves displayed in Figure 5 show the apparent viscosity of the CNT water based nanofluid as a function of shear after different resting times and an initial preshear stress rate of 10Pa. It is observed from figure 5 that all the curves are well superimposed indicating that the resting time has no effect on the rheological behaviour of the CNT water based nanofluid. This also reveals that the structural network of the nanosuspension does not evolve after cessation of preshear and that the nanofluid is stable within 1h after a high preshear of 10Pa.

As reported in the previous section, it is noted that the viscosity of the nanosuspension at high shear rate is not affected by the resting time. Actually, the high shear viscosity reported in Figure 4 and 5 tends to a value of 5.8mPa.s which is in agreement with the value obtained from figures 2 and 3.

## **Conclusion**

A detailed study on the effect of a well-controlled preshear history on the rheological properties of CNT water based nanofluid has been performed. The results indicate that the preshearing caused the breakdown in the structural network of the nanofluid, due to the presence of agglomerates, following the rate of pre-shear. It was shown that the viscoelastic behavior of the nanofluid at low shear rate is strongly dependent on the shear stress history, as well as the shear-thinning behavior of the nanofluid at higher shear rate. Then, it was reported that the high shear viscosity of the nanofluid is enhanced due to the presence of aggregates. The maximum size of aggregates was predicted and favorably compared to SEM characterization of the nanofluid. Finally, it was shown that the structural network of highly pre-sheared nanosuspension is stable and does not evolve at rest. The decrease in pre-shear rate leads to a structural network of the nanosuspension which reforms at rest following their disruption. The results of this study also show the relevance of the rheological characterization concerning structural information of nanofluids and reveal the time-dependent behavior of the studied nanofluid.

## **Conflict of Interest**

None

## Acknowledgments

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## References

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## **Figure Captions**

Fig 1 SEM image taken from dried nanofluid

Fig 2 Effect of preshear stress rate (PS) on the viscosity of the CNT nanofluid

Fig 3 Comparison of experimental and predicted values of viscosity – influence of aggregates

Fig 4 Effect of resting time (RT) on the viscosity of the CNT nanofluid after a preshear of 5Pa for 60s

Fig 5 Effect of resting time (RT) on the viscosity of the CNT nanofluid after a preshear of 10Pa for 60s

## **Table Captions**

Table 1 Properties of nanotubes

Table 2 Properties of the nanofluid



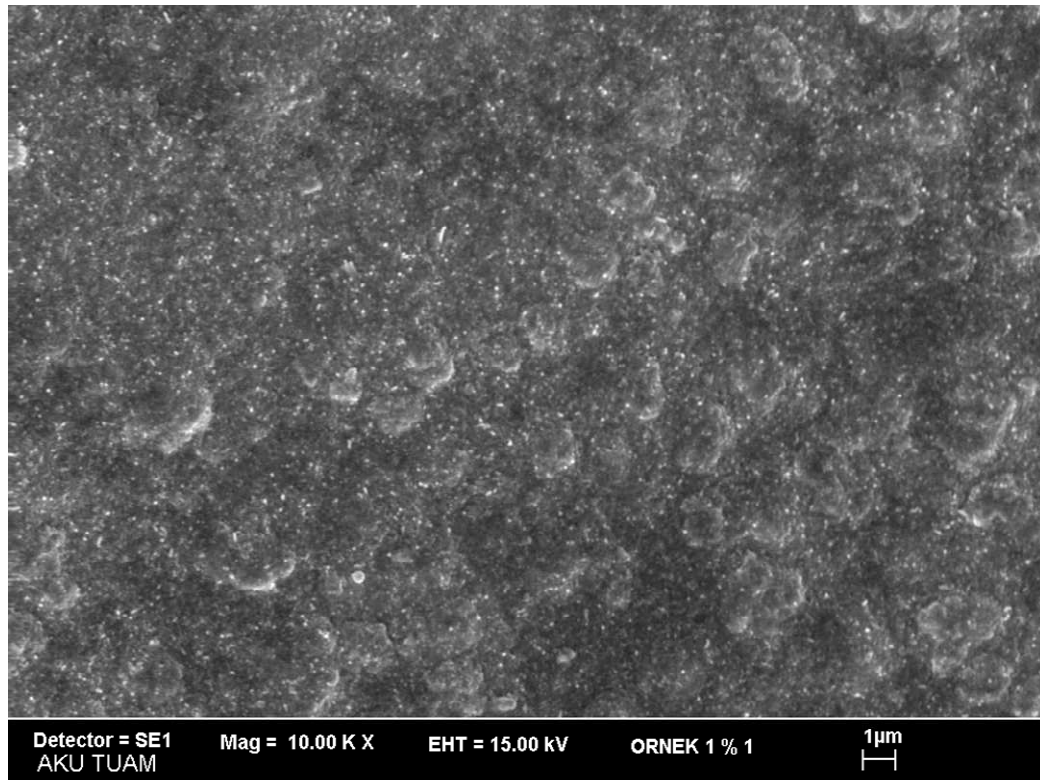


Fig 1 SEM image taken from dried nanofluid

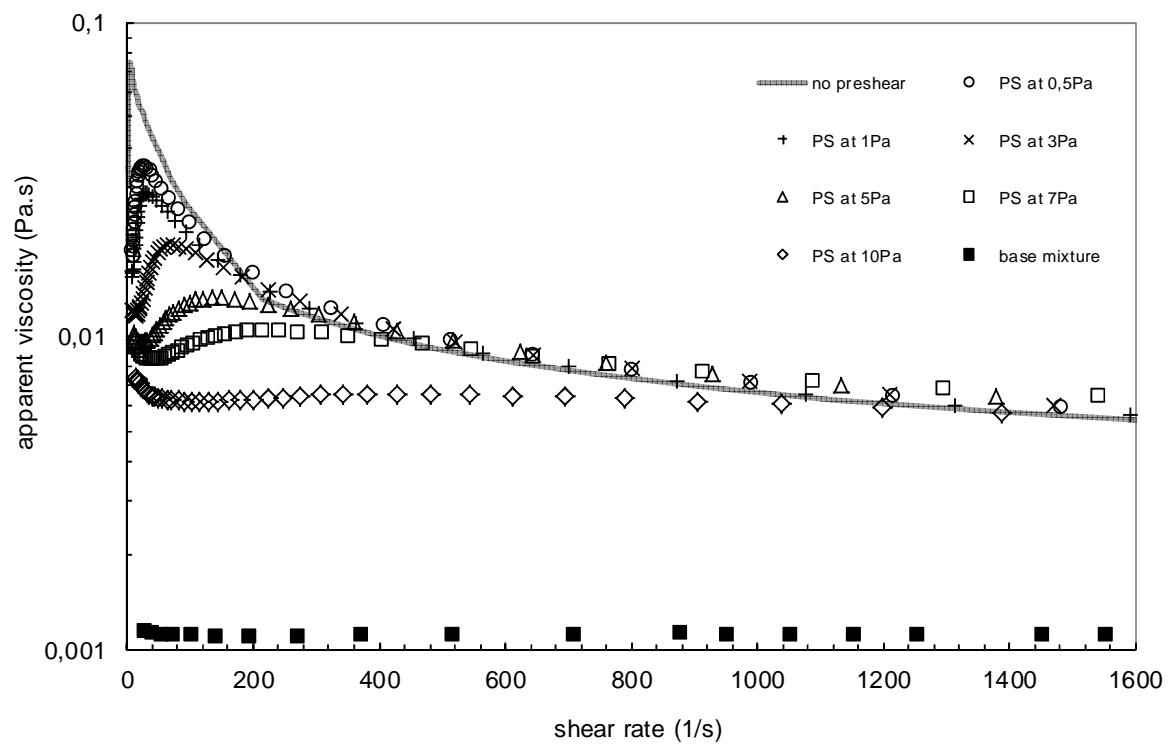


Fig 2 Effect of preshear stress rate (PS) on the viscosity of the CNT nanofluid

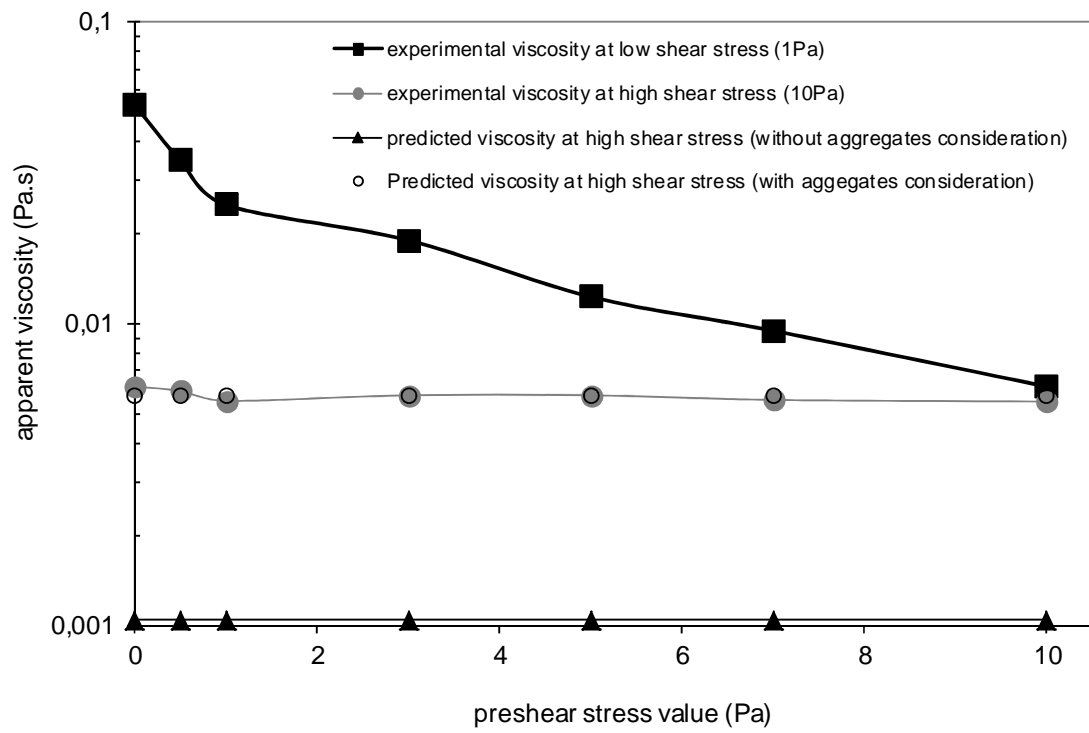


Fig 3 Comparison of experimental and predicted values of viscosity – influence of aggregates

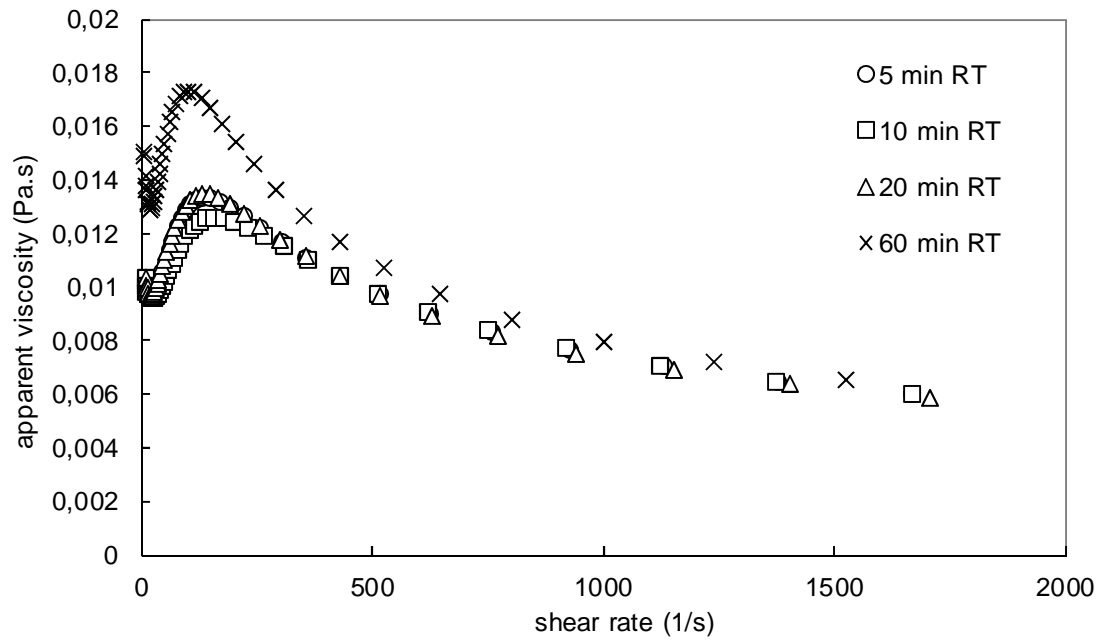


Fig 4 Effect of resting time (RT) on the viscosity of the CNT nanofluid after a preshear of 5Pa for 60s

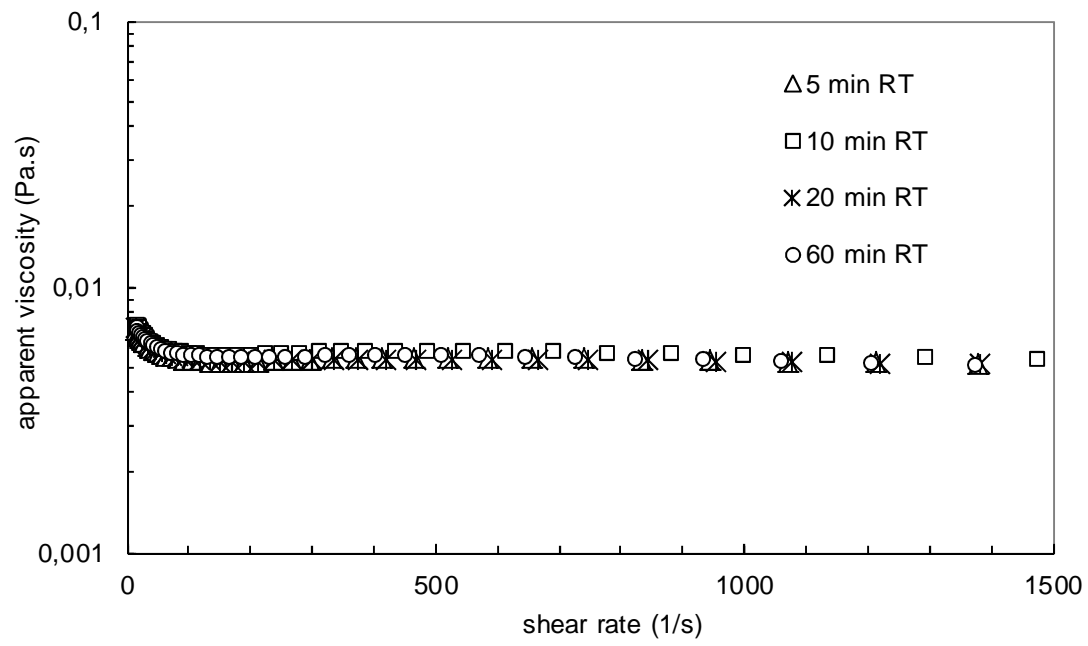


Fig 5 Effect of resting time (RT) on the viscosity of the CNT nanofluid after a preshear of 10Pa for 60s

Table 1 Properties of nanotubes

Nanoparticles	Average length ( $\mu\text{m}$ )	Average diameter (nm)	Density ( $\text{kg/m}^3$ )
Carbon Nanotubes	1.5	9.2	1800

Table 2 Properties of the nanofluid

Nanofluid	Nanotubes weight fraction (%)	Nanotubes volume fraction (%)	SDBS weight fraction (%)
Aqueous Carbon Nanotubes suspension	1	0.55	2